

Electrochemical/Fe Dual-Catalyzed Perfluoroalkylation-Thiolation of Alkenes

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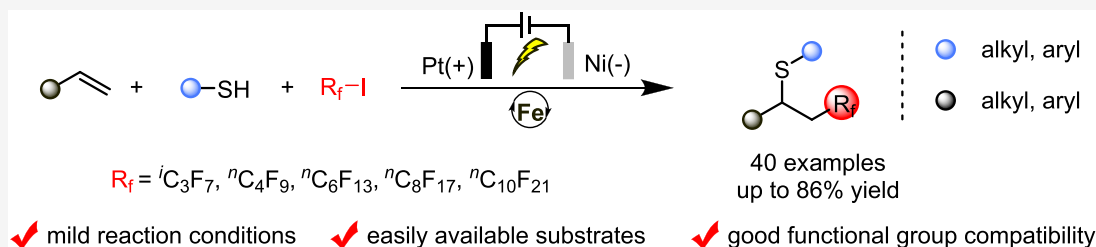
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ABSTRACT: A novel electrochemical/Fe dual-catalyzed perfluoroalkylation-thiolization of alkenes under mild conditions has been developed. This protocol utilizes commercially available reagents, cheap electrodes, and simple equipment. Diverse polyfunctionalized perfluoroalkyl-substituted derivatives were successfully obtained in a direct and efficient way with a broad substrate scope and excellent functional group tolerance. Notably, the gram-scale synthesis and late-stage modification of biorelevant compounds highlighted the potential application of the strategy. Mechanistic studies such as cyclic voltammetry test, free radical trapping experiments, and control experiments suggest that this strategy involves the generation of a fluorinated radical and simultaneously undergoes addition to alkenes followed by iron-mediated C–S bond formation.

INTRODUCTION

Owing to the unique chemical properties, the incorporation of fluorine-containing moieties into drug molecules can significantly improve the metabolic stability, lipophilicity, and biological activity of drugs.¹ Especially, more and more drugs with fluoroalkyl groups were developed,² like Miebo³ and Fulvestrant (Scheme 1a).⁴ In recent years, significant efforts have been devoted to introducing perfluoroalkyl motifs to compounds with various fluoroalkylation reagents.⁵ Among them, difunctionalization of an alkene is regarded as a convenient and straightforward tool capable of selectively forming two different chemical bonds.⁶ The metal-/photo-catalyzed fluoroalkylative alkene difunctionalization,⁷ such as carbonization,⁸ hydroxylation,⁹ hydrogenation,¹⁰ halogenation,¹¹ azidation,¹² and thio(seleno)cyanation,¹³ has been well developed. From a formal point of view, more novel strategies are still highly desirable in order to broaden the scope of functional group implantation in C=C bonds.

Furthermore, sulfur-containing compounds constitute essential structural motifs in drug development, owing to their versatile chemical reactivity and significant biological activities,¹⁴ including sulfonamides and sulfides (e.g., Nelfinavir)¹⁵ (Scheme 1a). Therefore, the simultaneous installation of fluorine and sulfur groups will be an attractive strategy to construct complex molecules and potentially improve their bioactive properties.¹⁶ However, only a few fluoroalkylation-thiolation reactions of alkenes to install both C–F and C–S bonds have been reported. In 2018, Song developed a copper-

catalyzed intermolecular reductive radical difluoroalkylation-thiolation of aryl alkenes.¹⁷ Meanwhile, Cai's group reported a difluoroalkylation-thiolation of alkenes with $\text{BrCF}_2\text{CO}_2\text{Et}$ and thiols by iron-facilitated visible light (Scheme 1b).¹⁸ Very recently, Liu et al. realized a photoinduced radical 1,2-difunctionalization of various alkenes with perfluoroalkyl iodides and thiosulfonates enabled by the electron donor–acceptor complex.¹⁹ In spite of the usefulness of these methods, issues including the need to use costly catalysts and complicated synthesis of substrates remain. So, it is necessary to develop a simpler and efficient approach to thiofluoroalkylation of alkenes with easily available fluoro/sulfur sources.

Organic electrosynthesis has the advantages of mild conditions, no need for excessive oxidation reagents, and environmental benignity making it widely used in chemical synthesis and other fields.²⁰ With the rise of organic electrosynthesis, many researchers have used this promising technology to introduce fluoroalkyl groups in small molecules.²¹ Herein, inspired by these contributions and our

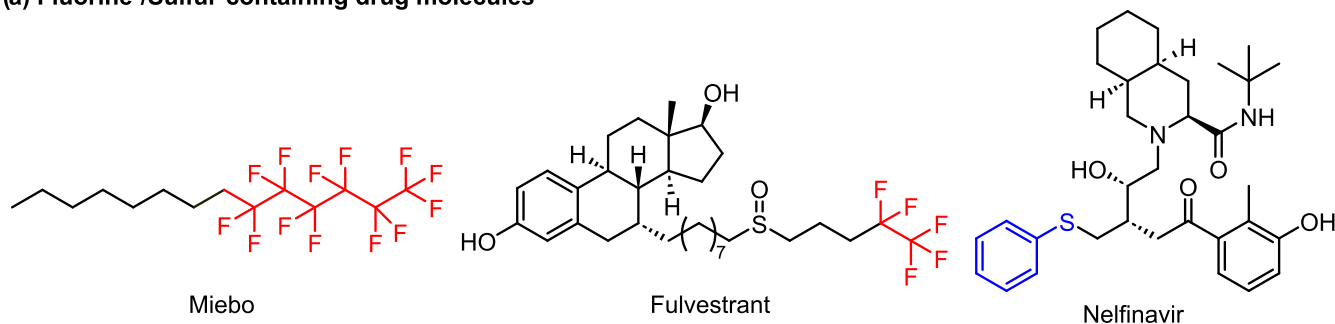
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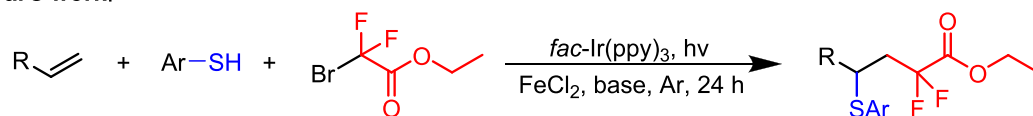
Scheme 1. Methods for the Synthesis of S-Fluoroalkyl Alkene

(a) Fluorine-/Sulfur-containing drug molecules

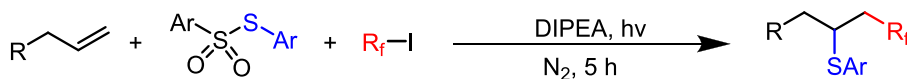


(b) Previous works

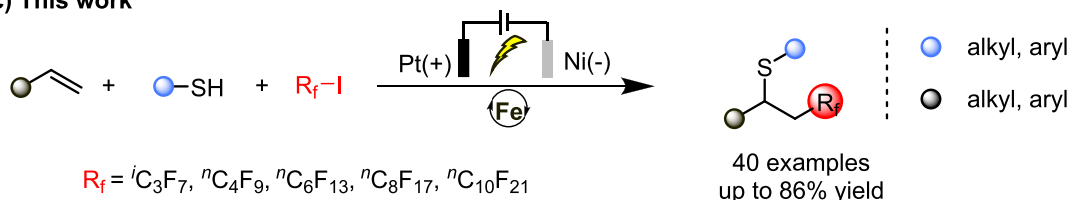
Cai's work:



Liu's work:



(c) This work



✓ mild reaction conditions ✓ easily available substrates ✓ good functional group compatibility

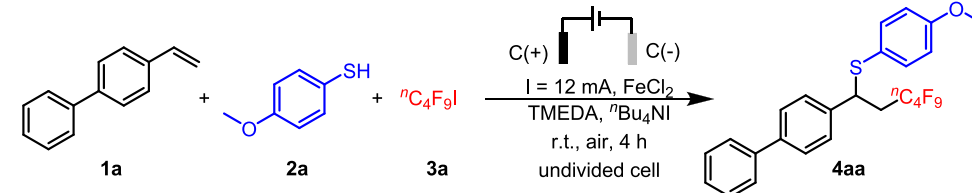
interest in constructing C–S bonds,²² we envision an electrochemical/Fe dual-catalyzed perfluoroalkylation-thiolation of alkenes with perfluoroalkyl iodides and free thiols to the simultaneous installation of perfluoroalkyl and sulfur-containing moieties (Scheme 1c).

RESULTS AND DISCUSSION

In the initial studies, 4-vinylbiphenyl **1a**, 4-methoxybenzenethiol **2a**, and perfluorobutyl iodide **3a** were selected as the model substrates to optimize the reaction conditions (Tables 1 and S1–S8). Initially, different kinds of solvents, such as MeOH, MeCN, DMSO, and toluene, were tested, but all of them gave disappointing yields (entries 2–5). In 2022, Chen et al. demonstrated that H₂O could promote the production of perfluoroalkyl radicals.²³ On this basis, the ratio of water was screened (entries 1, 6–10),²⁴ finally DCE/H₂O (v/v = 1:7) was chosen as the optimal solvent with a yield of 65%. Catalyst screening indicated that FeCl₂ was indispensable for the reaction, and varying its equivalent did not enhance the yield (entries 11–13). Next, we evaluated organic and inorganic bases and found that TMEDA was essential for this protocol. Additionally, the reaction efficiency decreased significantly with either an increase or a decrease in the TMEDA loading

(entries 13–15). Then, an investigation of electrolytes, including ⁿBu₄NPF₆, ⁿBu₄ClO₄, KI, NaBr, was carried out, indicated that phase transfer catalysts were better than inorganic salts in terms of yield (entry 17). Further changes of the current to 2, 4, 8, or 16 mA revealed that 2 mA was a preferable choice to obtain product **4aa** in 73% yield (entry 18). At last, we tested a series of electrodes, and Pt(+)/Ni(–) proved to be the better match with 78% yield (Table 1, entry 19). These results indicate that the base and supporting electrolyte play crucial roles in this reaction, while electricity is essential.

Under the optimized conditions, the scope and compatibility of alkenes were first investigated, employing **2a** and **3a** as model substrates. As shown in Scheme 2, styrenes with electron-donating (phenyl, methyl, *tert*-butyl, methoxy, acetoxy) or electron-withdrawing (halide, trifluoromethyl) groups on the aryl ring were tolerated well, and could give the desired products **4aa–4al** in good yields (56–78%). When the methyl group was located at the *ortho*-, *meta*-, or *para*-position of the benzene ring, the yields of **4ac–4ad** showed minimal variation, demonstrating that this reaction was scarcely affected by the steric effect. α -Methylstyrene could also be applied to this reaction and delivered the corresponding product **4am** in 60%

Table 1. Optimization of the Reaction Conditions^a


entry	variation from the standard conditions	yield ^c
1	none	65
2	MeOH as solvent	5
3	MeCN as solvent	9
4	DMSO as solvent	trace
5	toluene as solvent	trace
6	DCM/H ₂ O (v/v = 2:6) as solvent	51
7	DCM/H ₂ O (v/v = 4:4) as solvent	46
8	DCM/H ₂ O (v/v = 6:2) as solvent	38
9	DCM/H ₂ O (v/v = 7:1) as solvent	33
10	DCE/H ₂ O (v/v = 1:7) as solvent	42
11	CuI, FeBr ₃ instead of FeCl ₂	ND ^d , trace
12	40, 80, 100% mmol of FeCl ₂	27, 31, 56
13	no TMEDA, no FeCl ₂ , or no current	ND
14	DBU, DABCO, Na ₂ CO ₃ instead of TMEDA	43, 32, 23
15	0.5, 1.0, 2.0 equiv of TMEDA	37, 45, 64
16	3, 5, 12 h instead of 4 h.	45, 63, 59
17	ⁿ Bu ₄ NPF ₆ , ⁿ Bu ₄ ClO ₄ , KI, NaBr instead of ⁿ Bu ₄ NI	57, 64, 38, 34
18	2, 4, 8, 16 mA instead of 12 mA	73, 70, 47, 44
19 ^b	C(+)/Ni(-), C(+)/Fe(-), Pt(+)/Ni(-), Pt(+)/Fe(-) instead of C(+)/C(-)	70, 68, 78, 74

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.5 mmol, 2.5 equiv), **3a** (0.6 mmol, 3.0 equiv), TMEDA (0.3 mmol, 1.5 equiv), FeCl₂ (0.06 mmol, 0.3 equiv), ⁿBu₄NI (0.2 mmol, 1.0 equiv), DCM (1.0 mL) and H₂O (7.0 mL), *I* = 12 mA, C(+)/C(-), under air, rt, 4 h. ^b*I* = 2 mA. ^cIsolated yields. ^dND = no detected.

yield. As delighted, 2-naphthylethylene and 1-naphthylethylene were applicable to the reaction, accomplishing the desired products **4an** and **4ao** with 76 and 73% yields, respectively. Furthermore, the reaction of 2-vinylpyridine gave the corresponding product **4ap** in 55% yield. Nonterminal olefins such as indene could react smoothly, generating the desired product **4aq** (54%). Notably, nonactive alkenes could react smoothly to form the desired products **4ar**, **4as**, and **4at** in 42, 45, and 38% yields, respectively.

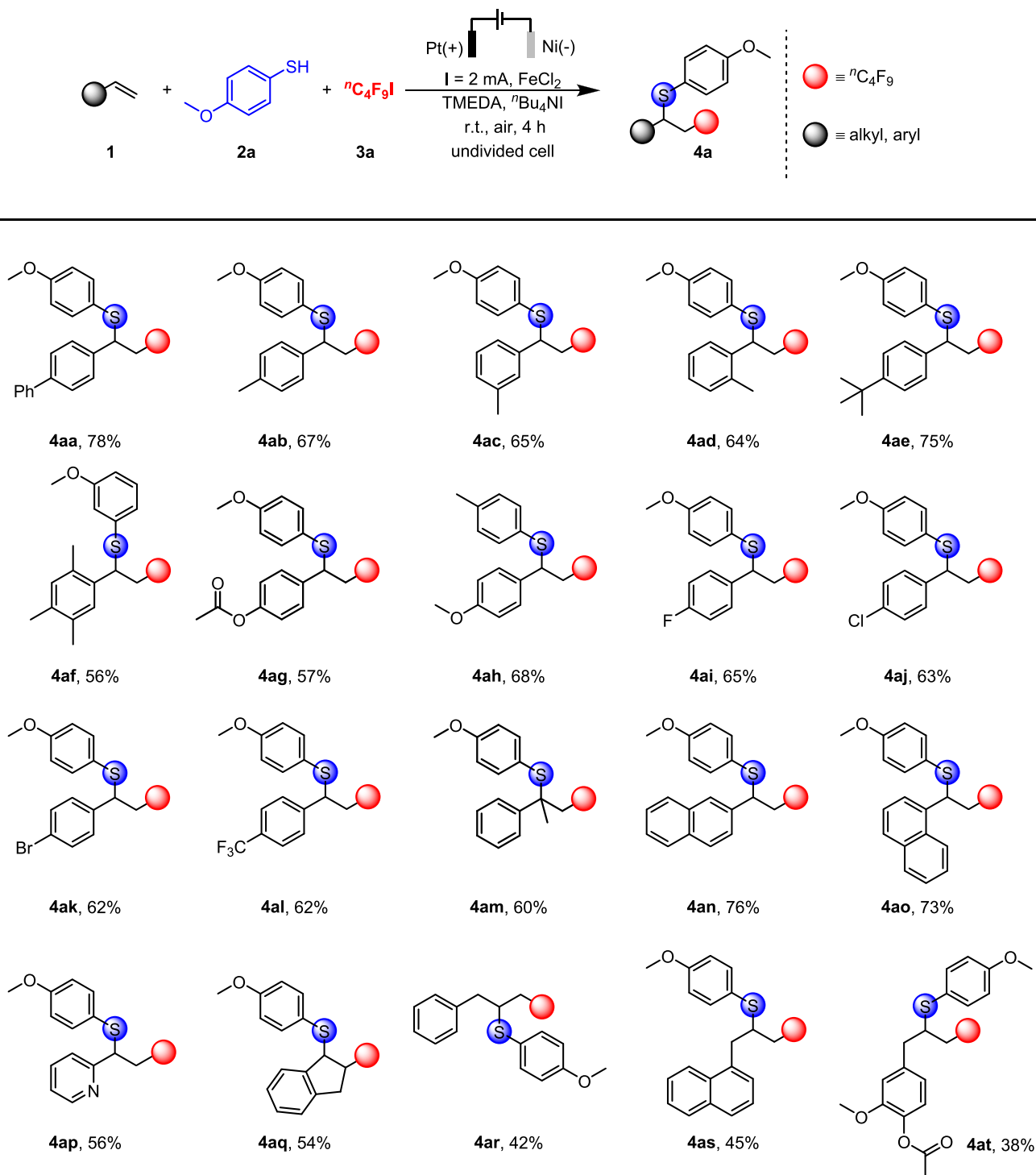
We next explored the scope of thiols (Scheme 3). Generally, phenylthiol substituted with functional groups, such as methoxyl, methyl, *iso*-propyl, *tert*-butyl, and halogen (F, Cl, Br), could be well tolerated to deliver the desired products in satisfactory yields (**4aa–4ia**, 68–86% yields), regardless of their positions on the aryl rings. Moreover, the reaction exhibited compatibility to disubstituted phenylthiols, generating the corresponding products **4ja–4la** with 76–83% yields. However, 1,3,5-triisopropylbenzenethiol gave a much lower yield of **4ma** (42%) compared with **4ea** (84%), indicating a steric effect from the bulky isopropyl groups. As delighted, 2-naphthalenyl thiol and 2-methyl-3-furanthiol were also effectively transformed into desired products **4na** (70%) and **4oa** (51%). Surprisingly, the cysteine derivative was successfully applied to obtain product **4pa** in 58% yield, which further expanded the application potential of this strategy. Furthermore, a range of perfluoroalkyl iodides, including ICF(CF₃)₂, ⁿIC₆F₁₃, ⁿIC₈F₁₇, and ⁿIC₁₀F₂₁, were examined and generated the desired products **4qa–4ta** in delightful yields (67–75%).

The gram-scale synthesis was performed on a 4 mmol scale, and 1.91 g of desired product **4aa** was isolated in 88% yield

(Scheme 4a). Additionally, **4aa** could be further oxidized to the sulfone derivative **A1** by *m*-CPBA with a yield of 95% (Scheme 4b). Notably, late-stage functionalization of bioactive molecules such as perospirone, estrone, piperonyl, and mentol proceeded efficiently, providing access to diversified analogues in practical yields (Scheme 4c).

Some preliminary mechanistic studies were then performed to gain insights into the mechanism of this protocol (Scheme 5). When the radical scavenger 2,2,6,6-tetramethylpiperidinoxy (TEMPO) was added under the optimized reaction conditions, the reaction was completely inhibited. Concurrently, the TEMPO-adduct product **B** was detected by liquid chromatography–mass spectrometry (LC–MS), which proved that this reaction might undergo radical pathways. Then, when **2a** was replaced by 4,4'-dimethoxydiphenyldisulfide under the standard conditions, the expected product **4aa** was not obtained. This result indicates that the disulfide compound functions as a byproduct rather than an intermediate. We failed to separate compound **C** when just **1a** and **3a** were submitted into the system, which indicated that compound **C** was possible not attended to the reaction process.

To further investigate the process of the perfluoroalkylation-thiolization of alkenes, a number of cyclic voltammetry tests were conducted (Figures 1 and S2). When **1a**, **2a**, and **3a** were added together, an obvious oxidation signal was observed compared with the background (grass green line, Figure 1). Moreover, there was a significant decrease in the oxidation potential of the reaction with the addition of FeCl₂ to the reaction mixture (from 3.43 to 3.23 V vs Ag/AgCl). It was

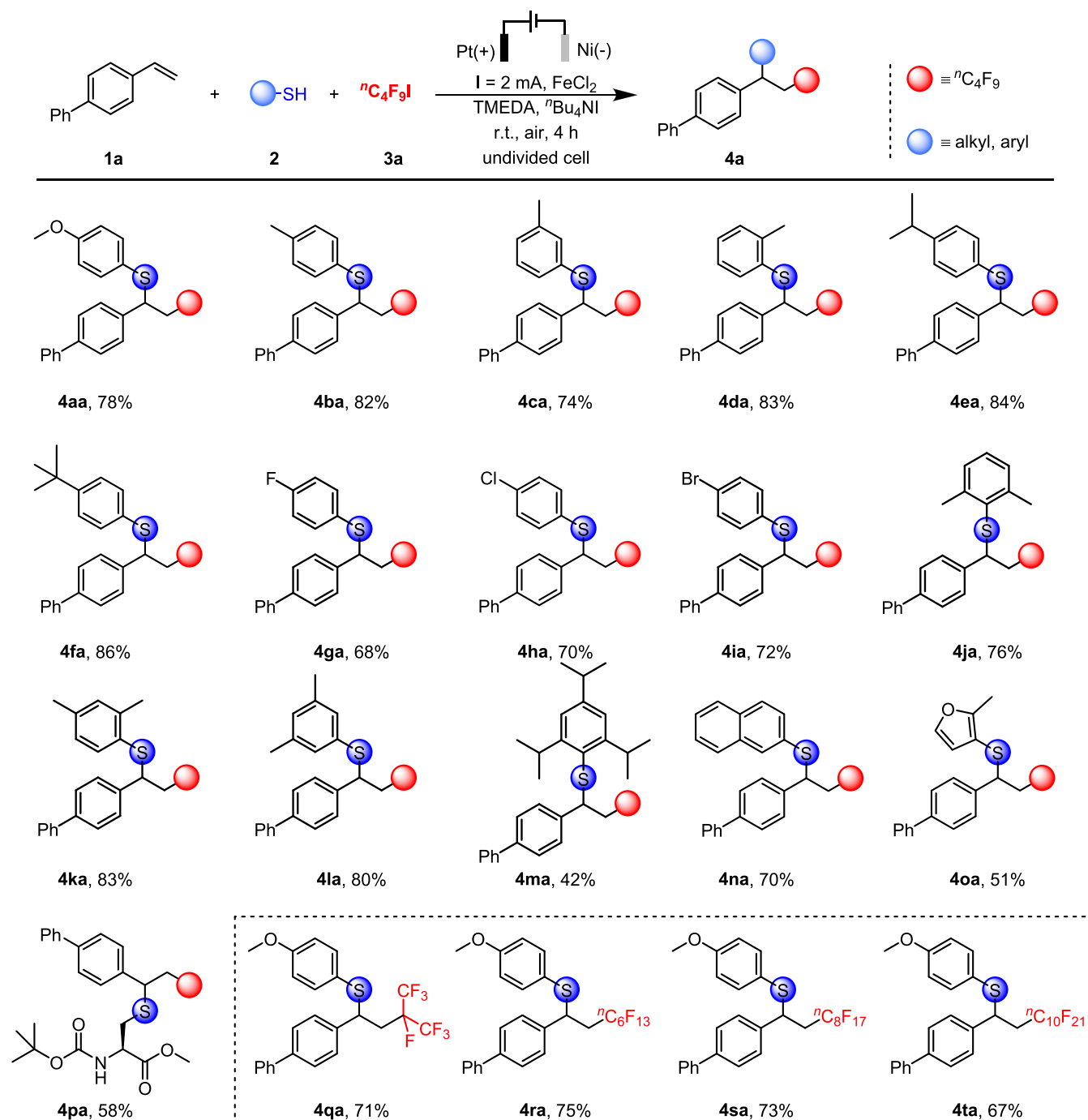
Scheme 2. Scope of Alkene Substrates^{a,b}

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.5 mmol, 2.5 equiv), **3a** (0.6 mmol, 3.0 equiv), TMEDA (0.3 mmol, 1.5 equiv), FeCl_2 (0.06 mmol, 0.3 equiv), $n\text{Bu}_4\text{NI}$ (0.2 mmol, 1.0 equiv), DCM (1.0 mL) and H_2O (7.0 mL), $I = 2 \text{ mA}$, Pt(+)/Ni(−), under air, rt, 4 h. ^bIsolated yields.

implied that FeCl_2 can decrease the reaction oxidation potential to promote the oxidation process of this protocol.

On the basis of mechanistic studies and relevant literature, a possible mechanism was proposed (Scheme 6). First, the halogen bonding interaction between perfluorobutyl iodide and either organic base (TMEDA) or water facilitates the generation of perfluoroalkyl radical **A**.^{23,25} Subsequently, perfluoroalkyl radical **A** undergoes an addition reaction with

alkene **1a** to form carbon radical intermediate **B**. On the other hand, **2a** reacts with FeCl_2 to generate intermediate **C** (Fe(II)),¹⁸ which combines with radical intermediate **1** and is subsequently oxidized to intermediate **D** (Fe(III)) on the anode. Finally, reduction of **D** occurs at the cathode to provide the target product **4aa** along with the regeneration of Fe^{2+} to accomplish the catalytic cycle.²⁶

Scheme 3. Scope of Thiophenol and Fluoroalkyl Iodide Substrates^{a,b}

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.5 mmol, 2.5 equiv), **3a** (0.6 mmol, 3.0 equiv), TMEDA (0.3 mmol, 1.5 equiv), FeCl₂ (0.06 mmol, 0.3 equiv), ⁿBu₄NI (0.2 mmol, 1.0 equiv), DCM (1.0 mL) and H₂O (7.0 mL), I = 2 mA, Pt(+)/Ni(−), under air, rt, 4 h. ^bIsolated yields.

CONCLUSIONS

In summary, the research introduces an innovative electrochemical/Fe dual-catalyzed three-component radical relay strategy, achieving a milestone in the perfluoroalkylation-thiolation of alkenes. This strategy is efficient, sustainable, and easily equipped and compatible with diverse substrates. Notably, late-stage modification of biorelevant compounds with gram-scale feasibility demonstrates promising synthetic utility. This approach demonstrates the efficiency of combining

electrocatalysis with FeCl₂ to address practical challenges in synthetic chemistry.

ASSOCIATED CONTENT

Data Availability Statement

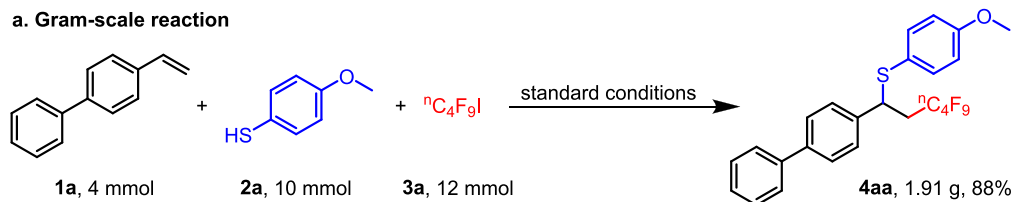
The data underlying this study are available in the published article and its [Supporting Information](#).

Supporting Information

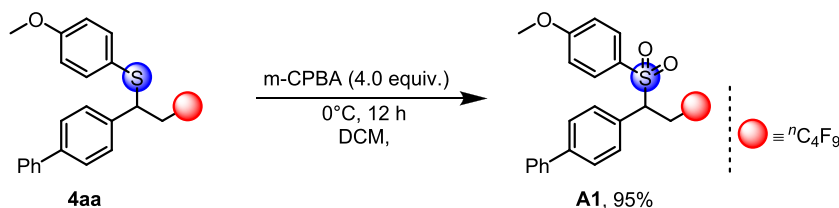
The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.joc.5c01984>.

Scheme 4. Synthetic Applications

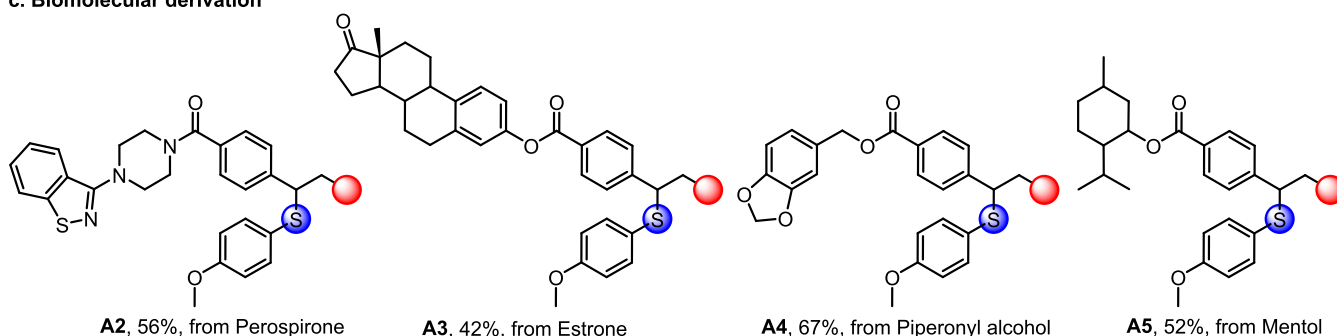
a. Gram-scale reaction



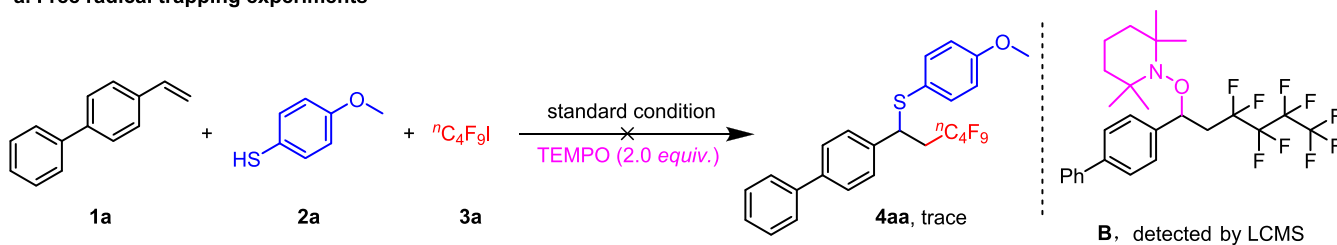
b. Sulfur-to-sulfonization of product 4aa



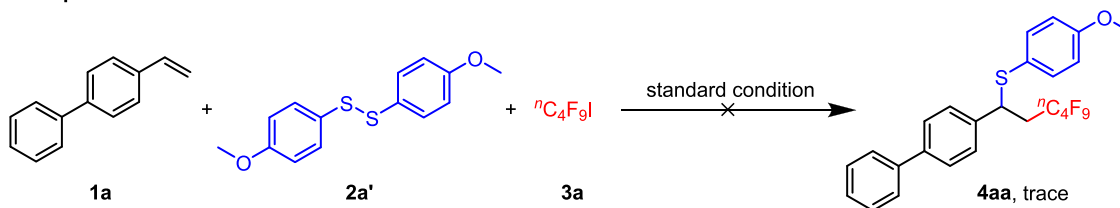
c. Biomolecular derivation

Scheme 5. Mechanistic Investigations^a

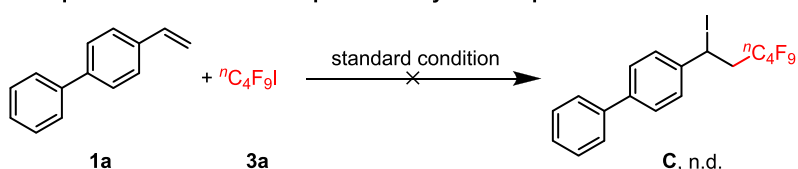
a. Free radical trapping experiments



b. Experimental verification of disulfide as intermediate



c. Experimental verification of perfluoroalkyl iodized product as intermediate



^aStandard conditions: 1a (0.2 mmol, 1.0 equiv), 2a (0.5 mmol, 2.5 equiv), 3a (0.6 mmol, 3.0 equiv), TMEDA (0.3 mmol, 1.5 equiv), FeCl₂ (0.06 mmol, 0.3 equiv), ⁿBu₄NI (0.2 mmol, 1.0 equiv), DCM (1.0 mL) and H₂O (7.0 mL), I = 2 mA, Pt(+)/Ni(−), under air, rt, 4 h.

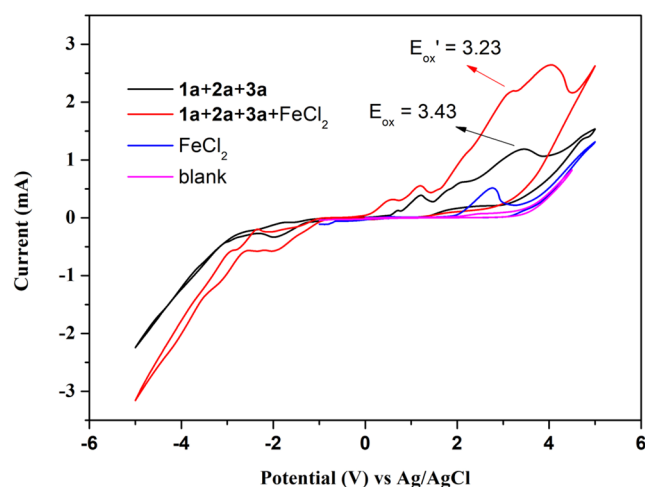
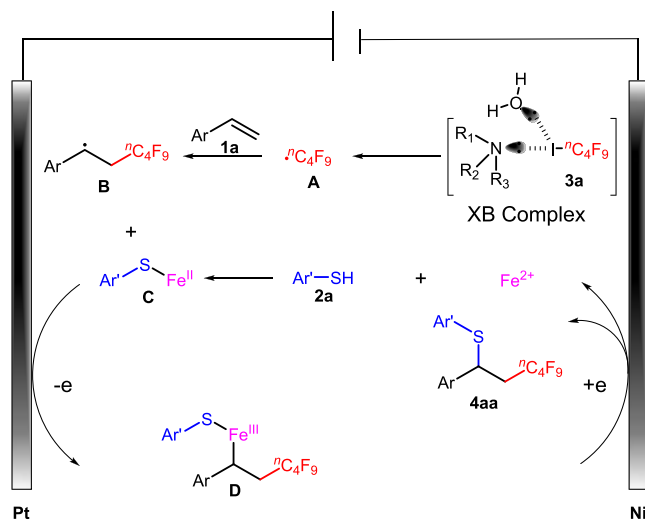


Figure 1. Cyclic voltammetry experiments.

Scheme 6. Proposed Mechanistic Pathway



Experimental procedures, reaction optimization, proposed mechanism, and spectral data for all new compounds (PDF)

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<https://pubs.acs.org/10.1021/acs.joc.5c01984>

Author Contributions

W.W.: Methodology, formal analysis, data curation, writing—original draft. K.Z.: Methodology, formal analysis. H.X.: Methodology. H.Z.: Methodology. W.C.: Writing—original draft, writing—review and editing, methodology, funding acquisition, supervision.

Notes

The authors declare no competing financial interest.

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